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Synthesis of 5-(trifluoromethyl)-5*H*-chromeno[3,4-*b*]pyridines from 3-nitro-2-(trifluoromethyl)-2*H*-chromenes and aminoenones derived from acetylacetone and cyclic amines

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ABSTRACT

Reactions of 3-nitro-2*H*-chromenes with aminoenones derived from acetylacetone and cyclic amines proceed diastereoselectively to give functionalized 2,3,4-trisubstituted chromanes as a result of nucleophilic addition of the vinylogous β-methyl group at the C-4 atom of the chromene system. From these compounds, under acidic conditions, 5-(trifluoromethyl)-5*H*-chromeno[3,4-*b*]pyridines and 4-acetoacetonyl-3-nitro-2-(trichloromethyl/phenyl)chromanes, depending on the substituent at the 2-position, were obtained in moderate to good yields.

Many derivatives of chromane (3,4-dihydro-2*H*-1-benzopyran) and 2*H*-chromene (2*H*-1-benzopyran) are natural compounds that are widely abundant in the plant kingdom.[1] Some of these, as well as a series of synthetic 2*H*-chromenes, have found use as pesticides and promising drugs.[2] 3-Nitro-2*H*-chromenes, as conjugated nitroolefins, possess unique chemical reactivity in both nucleophilic and cycloaddition reactions because of their reactive double bond. As a result, these compounds have attracted attention as excellent building blocks for the preparation of various more complex heterocyclic compounds.[3] In particular, 3-nitro-2*H*-chromenes react with diverse C-, N-, S-, and P-nucleophiles to give a wide range of substituted[4] and fused[5] chromanes. Recently, the reactions of 2-aryl-3-nitro-2*H*-chromenes with α,α-dicyanoolefins in the presence of triethylamine, leading to the preparation of 6-aryl-6*H*-dibenzo[*b*,*d*]pyran derivatives, were reported.[6] At the same time, to the best of our knowledge, very little information is available on the reactions of 3-nitro-2*H*-chromenes with push–pull enamines.[7] It has been reported that 2-aryl-3-nitro-2*H*-chromenes react with methyl β-methylaminocrotonate to give mixtures of the addition product, methyl 3-methylamino-2-(2-aryl-3-nitrochroman-4-yl)-2-butenoate, and 1-benzopyrano[3,4-*b*]pyrrole, formed via the Grob cyclization,[8] whereas their reactions with ethyl β-morpholinocrotonate took an entirely different course and gave ethyl 3-morpholino-4-(2-aryl-3-nitrochroman-4-yl)-2-butenoates.[9] The latter result belongs to a rare case, where the reaction proceeded not at the more nucleophilic α-position of an enamino ester, but at the vinylogous β-methyl group. This is of particular interest because it results in the formation of a functionalized chromane system, which has not been reported previously.

Within the framework of a research program on the synthetic opportunities offered by 3-nitro-2-trihalomethyl-2*H*-chromenes **1** for the preparation of organic molecules of potential interest in biomedicinal chemistry and materials science,[5] we examined the behavior of compounds **1** in reactions with the push–pull enamines derived from acetylacetone and cyclic amines, namely (*E*)-4-morpholino- and (*E*)-4-piperidinopent-3-en-2-ones, with the purpose of studying the structures and acid-catalyzed transformations of the addition products **2**. The present communication describes a new type of pyridine ring annulation, which consists of the conversion of compounds **2** into 5-(trifluoromethyl)-5*H*-chromeno[3,4-*b*]pyridines **4**, and demonstrates the dramatic effect of the trifluoromethyl group on the reaction pathway.

We found that the reaction of chromenes **1** with 4-morpholino- and 4-piperidinopent-3-en-2-ones in dry acetonitrile at room temperature for 2–48 h resulted in the stereoselective formation of 2,3,4-trisubstituted chromanes **2a**–**i** in 42%–83% yields as single diastereomeric products with *cis*–*trans*-configuration ($^3J_{H2,H3}$ = $^3J_{H3,H4}$ = 1.5 Hz) at the C(2)–C(3) and C(3)–C(4) bonds, respectively, and with *E*-configuration at the double bond (Scheme 1, Table 1).[10] The stereochemistry of the products was confirmed by an X-ray diffraction study of crystals of **2a** (Fig. 1).[11]

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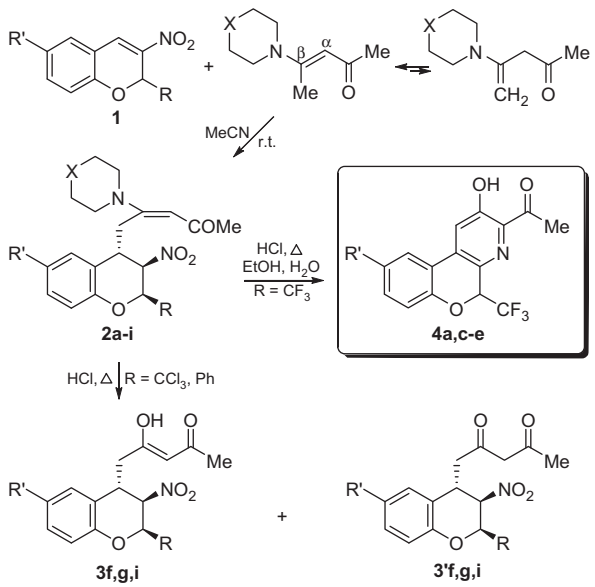


Scheme 1. Synthesis of compounds **2**–**4**.

Table 1
Synthesis of compounds **2**–**4**

R	R′	X	Product **2**	Time (h)	Yield (%)	Product **3**, **4**	Yield (%)
CF_3	H	CH_2	**2a**	2	71	**4a**	34
CF_3	H	O	**2b**	5	83	**4a**	33
CF_3	Br	O	**2c**	3	80	**4c**	46
CF_3	NO_2	O	**2d**	6	70	**4d**	42
CF_3	MeO	O	**2e**	48	64	**4e**	34
CCl_3	H	O	**2f**	30	42	**3f**[a]	56
CCl_3	Br	CH_2	**2g**	7	56	**3g**[b]	63
CCl_3	Br	O	**2h**	21	52	**3g**	66
Ph	H	O	**2i**	7	62	**3i**[c]	60

[a] **3f**:**3′f** = 88:12.
[b] **3g**:**3′g** = 91:9.
[c] **3i**:**3′i** = 69:31.

The reaction time varied according to the nature of the chromene molecule. In general, 2-CF_3-chromenes were more reactive than 2-CCl_3- and 2-Ph-chromenes; conjugate addition of an enamine to a CF_3-chromene containing an electron-donating MeO group required a longer period of time for completion of the reaction, while electron-withdrawing groups (Br, NO_2) required relatively short reaction times. Addition of push–pull enamines at the vinylogous β-Me group possibly results from the *E*-configuration of the starting enamines, which hinders the approach of the α-C atom to the activated double bond of the chromene system.

Next we examined the acid hydrolysis of enamino ketones **2a**–**i** in aqueous ethanol at reflux in the presence of concentrated HCl. As expected, hydrolysis of compounds **2f**–**i** successfully removed the amino function to give interesting acetoacetonyl derivatives **3f**, **g**, and **i** as a mixture of keto-enol tautomers **3** and **3′** in 56%–66% yields, with the same configuration (9%–31% of diketo form **3′**). As for the 2-CF_3-chromanes **2a**–**e**, there was a marked difference in their reactivity toward acid hydrolysis. Under the same conditions, instead of the corresponding acetoacetonyl derivatives **3**, we obtained 5-(trifluoromethyl)-5*H*-chromeno[3,4-*b*]pyridines **4a, c**–**e** in 33%–46% isolated yields (Scheme 1, Table 1). The structures of compounds **3** and **4** were established by IR, ^{1}H, ^{19}F, and ^{13}C NMR spectroscopy as well as by elemental analysis.[12]

These results indicate that the nature of the substituent at the 2-position influences the course of the acid-catalyzed hydrolysis of **2**, which in the case of a CF_3 group took an unexpected direction compared with the reaction of nonfluorinated chromanes. It is important to note that this new cyclization process tolerates both electron-donating (MeO) and electron-withdrawing (Br, NO_2) substituents on the benzene ring. Moreover, unlike previously known approaches for the synthesis of quinolines from nitro derivatives,[13] this reaction does not require any reducing agent or chromatographic purification of the final products, and thereby greatly facilitates the preparation of the target chromenopyridines **4**.

We can assume that cyclization of **2** into **4** was possible due to the high C–H acidity of the H-2 in *aci*-form **A**, which could be easily abstracted by the base to form **B** (nitrone–*N*-hydroxyenamine prototropic tautomeric equilibria through a [1,4]-H shift[14]), followed by dehydration of intermediate **B** into the corresponding α-nitrosoalkene **C**. Intramolecular nucleophilic attack of the side-chain enol on the nitroso group followed by dehydration results in the intermediate **D**, which undergoes a double [1,3]-H shift to give chromenopyridines **4** (Scheme 2). The lack of such reactivity in the case of compounds **2f**–**i** was apparently due to the lower acidity of the H-2 proton. It should be noted that a similar [1,4]-H shift has been previously observed by us in the spontaneous ring-contraction–rearrangement of CF_3-containing 1,2-oxazine *N*-oxides into 1-pyrroline *N*-oxides.[15]

The main information for the characterization of compounds **4** was obtained from the ^{1}H NMR spectra, which showed no methylene signal, a result consistent with the pyridine structure. The most downfield shifted signal was assigned to the hydroxyl proton, which appeared as a singlet at δ 12.0 in $CDCl_3$, the singlet

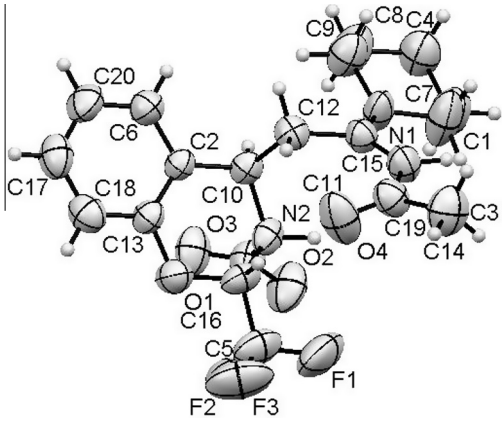
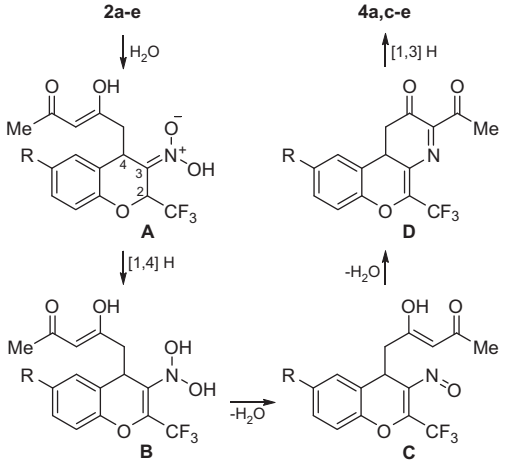


Figure 1. X-ray crystal structure of *cis*–*trans*-**2a** (ORTEP drawing, 50% probability level).



Scheme 2. Mechanism of the formation of chromeno[3,4-*b*]pyridines **4**.

at δ 7.58–7.77 was due to the resonance of the pyridine H-1 proton; assignment of all the signals was achieved by 2D $^{1}H–^{13}C$ HSQC, and HMBC experiments. In the 2D $^{1}H–^{15}N$ HMBC spectrum of **4c**, a signal was observed at δ 316.9 from liquid NH_3, indicating that the N was present in a pyridine ring.[16] In addition, the CF_3 group in the ^{19}F NMR spectra of chromenopyridines **4** manifested itself as a doublet at δ 85.0–85.5 (J = 7.0–7.3 Hz).

In conclusion, we have shown, for the first time, that the reaction of 3-nitro-2*H*-chromenes with push–pull enamines affords linear enamines with the chromanyl moiety, acid hydrolysis of which gave either 5*H*-chromeno[3,4-*b*]pyridines or 4-(acetoacetonyl)chromanes, depending on the nature of the substituent at C-2. When this substituent was a CF_3 group, a novel synthesis of fused pyridines followed, while acid hydrolysis of CCl_3- and Ph-containing derivatives proceeded in the expected manner to give interesting polyfunctional chromane derivatives. Further studies on the synthetic scope of these reactions are now in progress.

Acknowledgment

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10. General procedure for the synthesis of chromanes **2**. A mixture of chromene **1** (1.0 mmol) and (*E*)-4-morpholino(piperidino)pent-3-en-2-one (0.17 g, 1.0 mmol) in dry MeCN was stirred at room temperature for several hours (Table 1). The solid that formed was filtered and recrystallized from CH_2Cl_2/hexane (1:2) to give compound **2** as a white powder.
(*E*)-5-[(2*R**,3*R**,4*S**)-4-Morpholino-3-nitro-2-(trifluoromethyl)-3,4-dihydro-2*H*-chromen-4-yl]-3-penten-2-one (**2b**). Yield 0.35 g (83%), mp 164–165 °C (dec.). IR (KBr): 1655, 1632, 1549, 1490, 1448, 1374 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.13 (s, 3H, Me), 2.75 (dd, 1H, C*H*H, J = 14.6, 2.6 Hz), 3.30 (dt, 2H, N(CHH)$_2$, J = 12.9, 4.9 Hz), 3.30–3.40 (m, 3H, H-4, N(CH*H*)$_2$), 3.76 (t, 4H, O(CH$_2$)$_2$, J = 4.9 Hz), 4.23 (br t, 1H, CH*H*, J = 13.3 Hz), 5.16 (br s, 1H, H-3), 5.27 (br q, 1H, H-2, J = 6.0 Hz), 5.47 (s, 1H, =CH), 7.02–7.10 (m, 2H, H-6, H-8), 7.17 (br d, 1H, H-5, J = 7.3 Hz), 7.22–7.30 (m, 1H, H-7);^{13}C NMR (126 MHz, $CDCl_3$) δ 32.0, 32.5, 38.6, 47.0, 66.2, 70.7 (q, C-2, $^{2}J_{C,F}$ = 34.1 Hz), 77.6, 101.8, 117.6, 120.7, 122.3 (q, CF_3, $^{1}J_{C,F}$ = 280.8 Hz), 122.8, 128.3, 128.9, 152.0, 158.3, 196.5 (C=O); ^{19}F NMR (376 MHz, $CDCl_3$) δ 86.8 (d, CF_3, J = 6.0 Hz). Anal. calcd for $C_{19}H_{21}F_3N_2O_5$: C, 55.07; H, 5.11; N, 6.76. Found: C, 54.90; H, 4.97; N, 6.82.
11. X-ray diffraction study of compound **2a**. Diffraction data were collected at 295 K on an Xcalibur 3 automatic single-crystal diffractometer (graphite-monochromated MoKα radiation, ω-scans). The structure was solved by direct method and refined by the full-matrix least-squares method using the SHELX-97 program package.[17] The H atoms were located geometrically using the riding model. *Crystal data for* **2a**: $C_{20}H_{23}F_3N_2O_4$, M = 412.40, monoclinic crystals, space group $C2/c$, a = 19.979(2), b = 10.1081(8), c = 20.1833(19) Å, $\alpha = \gamma$ = 90.00, β = 96.442(8)°, V = 4050.2(6) Å^3, Z = 8, d_{calcd} = 1.353 g/cm^3, μ = 0.112 mm^{-1}, $F(000)$ = 1728. Crystallographic data for compound **2a** (CCDC 924323) have been deposited at the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK.
12. General procedures for the synthesis of compounds **3** and **4**. The corresponding chromane **2** (1.0 mmol) was refluxed in a mixture of EtOH (4 mL), H_2O (2 mL), and conc. HCl (0.2 mL) with vigorous stirring for 6 h. The reaction mixture was cooled to room temperature and the solid that formed was filtered, washed with H_2O (2 × 1 mL), dried at 70 °C, and recrystallized from CH_2Cl_2/hexane (1:2) (for **3**, a white powder) or MeOH (for **4**, colorless needles).
(2*R**,3*R**,4*S**)-1-[6-Bromo-3-nitro-2-(trichloromethyl)-3,4-dihydro-2H-chromen-4-yl]-2,4-pentanedione (**3g**). Yield 0.30 g (63%), mp 140–141 °C. IR (KBr): 1715, 1626, 1601, 1552, 1480, 1412, 1366 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) enol **3g** (91%) δ 2.11 (s, 3H, Me), 2.57 (dd, 1H, C*H*H, J = 16.4, 10.7 Hz), 2.90 (dd, 1H, CH*H*, J = 16.4, 4.2 Hz), 3.87 (dd, 1H, H-4, J = 10.7, 4.2 Hz), 4.50 (br s, 1H, H-3), 5.53 (s, 1H, H-2), 5.71 (s, 1H, =CH), 7.01 (d, 1H, H-8, J = 8.8 Hz), 7.33 (d, 1H, H-5, J = 2.2 Hz), 7.38 (dd, 1H, H-7, J = 8.8, 2.2 Hz); diketo **3′g** (9%) δ 2.76 (s, 3H, Me), 2.95–3.06 (m, 2H, CH_2), 3.63 (d, 1H, C*H*H, J = 15.8 Hz), 3.70 (d, 1H, CH*H*, J = 15.8 Hz), 3.96 (dd, 1H, H-4, J = 8.2, 5.5 Hz), 4.51 (br s, 1H, H-3), 5.61 (d, 1H, H-2, J = 1.5 Hz), 7.00 (d, 1H, H-8, J = 8.9 Hz), 7.28 (d, 1H, H-5, J = 2.3 Hz), 7.33 (dd, 1H, H-7, J = 8.9, 2.3 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) enol **3g** δ 24.2, 36.9, 45.6, 78.8, 80.5, 95.2, 100.6, 115.4, 119.1, 122.9, 131.3, 131.8, 151.8, 189.9, 190.1; diketo **3′g** δ 31.1, 35.3, 50.1, 56.9, 78.7, 80.6, 100.5, 115.3, 119.2, 122.8, 131.1, 131.7, 151.9, 200.2, 202.3. Anal. calcd for $C_{15}H_{13}BrCl_3NO_5$: C, 38.05; H, 2.77; N, 2.96. Found: C, 37.75; H, 2.59; N, 2.95.
1-{9-Bromo-2-hydroxy-5-(trifluoromethyl)-5H-chromeno[3,4-b]pyridin-3-yl}ethanone (**4c**). Yield 0.18 g (46%), mp 189–190 °C. IR (KBr): 1657, 1601, 1484, 1399, 1264, 1221 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.76 (s, 3H, Me), 5.60 (q, 1H, H-5, J = 7.1 Hz), 7.00 (d, 1H, H-7, J = 8.8 Hz), 7.47 (dd, 1H, H-8, J = 8.8, 2.1 Hz), 7.58 (s, 1H, H-1), 7.79 (d, 1H, H-10, J = 2.1 Hz), 11.93 (s, 1H, OH); ^{13}C NMR (126 MHz, $CDCl_3$) δ 25.7 (Me), 75.2 (q, C-5, $^{2}J_{C,F}$ = 31.6 Hz), 115.8 (C-9), 118.7 (C-1), 119.6 (C-7), 119.8 (C-10a), 123.0 (q, CF_3, $^{1}J_{C,F}$ = 286.9 Hz), 127.1 (C-10), 130.9 (C-10b), 134.8 (C-4a), 135.1 (C-8), 135.6 (C-3), 151.6 (C-6a), 159.6 (C-2), 206.5 (C=O); ^{19}F NMR (471 MHz, $CDCl_3$) δ 85.2 (d, CF_3, $J_{F,H}$ = 7.1 Hz); ^{15}N NMR (51 MHz, $CDCl_3$) δ 316.9; MS (EI): m/z 389 [^{81}Br, M+1]$^+$ (32), 387 [^{79}Br, M+1]$^+$ (32), 320 [M+1–CF_3]$^+$ (100), 318 [M−1–CF_3]$^+$ (100), 290 [M+1–CF_3–CO]$^+$ (18), 196 (17), 169 (13), 140 (19), 113 (24), 63 (21), 43 [Ac]$^+$ (79). Anal. calcd for $C_{15}H_9BrF_3NO_3$: C, 46.42; H, 2.34; N, 3.61. Found: C, 46.18; H, 2.48; N, 3.55.
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